EXPERIMENTAL INVESTIGATION OF ULTRA-HIGH VACUUM ADHESION AS RELATED TO THE LUNAR SURFACE

NINTH QUARTERLY PROGRESS REPORT

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ABSTRACT

Studies during this quarter have been concerned with measurements of adhesion between various silicate minerals whose surfaces have been formed in ultra-high vacuum by cleavage. The adhesion force has been investigated as a function of time at and degree of vacuum. It has been found that in most cases vacuum cleavage produces considerable surface electrostatic charging. Also, indications were found that the normal silicate atomic bonding forces were contributing to the adhesion. In all cases, the magnitude of the adhesion was considerably greater than that found for air-formed surfaces.

At present, the mechanism(s) responsible for the surface charging is not known. Possibilities are that it is produced by breakage of the atomic bonds resulting in electron deficiencies and excesses (this could be a random or non-random effect), that it is caused by charged dislocation migration under the permanent strains produced during cleavage, or that it could be a pseudo-piezoelectric effect.

Many more data are required before the processes acting to produce the observed adhesion can be determined with certainty.

1.0 INTRODUCTION

1.1 General

This report presents a summary of work accomplished during the period July 1, 1966 through September 30, 1966 on the study of the ultra-high vacuum frictional-adhesional behavior of silicates as related to the lunar surface. This work is being conducted for the Office of Advanced Research and Technology, National Aeronautics and Space Administration, under contract NAS 7-307.

Items of note during this quarter are first the addition of Dr. J. J. Grossman to the staff and, second, the publication in the September 15, 1966, issue of the Journal of Geophysical Research of some of the results obtained to date. Dr. Grossman received his degree in Physical Chemistry and has worked for a number of years in the fields of Surface Chemistry and Solid State Physics. He will be concerned with theoretical interpretation of the adhesion data as well as helping to guide the course of the experimentation.

1.2 Purpose and Importance of the Program

The primary purpose of this program is to obtain quantitative experimental data concerning the ultra-high vacuum adhesional-frictional behavior of the materials which may presently exist at the lunar surface (believed to be primarily silicates), and to obtain similar data for adhesion between those lunar surface materials and other engineering materials which may be placed upon the surface. Additional purposes of this program are to analyze these data with regard to the possible reactions of granular lunar materials to engineering operations, and to investigate means by which any problems posed by these reactions may be minimized.

The importance of this program is that adhesional-frictional phenomena may pose serious problems to lunar surface operations.

1.3 Approach

The approach used during this quarter has been to produce the silicate surfaces at ultra-high vacuum and measure the resultant adhesion force as a function of time at vacuum and degree of vacuum. The vacuum-formed surfaces have been used since they are representative of a possible upper bound to the range of lunar adhesional phenomena.

2.0 INSTRUMENTATION

The instrumentation used during this quarter was essentially the same as that reported for the previous quarter (Ryan, 1966). In summary, the pumping system consists of a bank of three sorption pumps and a 200 l sec^{-1} ion pump. The chamber is of 304 stainless steel. Pressure is monitored with a nude Bayard-Alpert gage. Adhesion force is measured by means of a mechanical spring with the spring extension determined by a cathetometer. Cleavage is obtained by wedging the sample apart in the vicinity of a previously cut notch.

3.0 EXPERIMENTAL DATA

3.1 Gas Bursts During Cleavage

It was reported in the previous quarterly report that a gas burst occurred during each cleavage. At times this was sufficient to raise the pressure into the 10^{-8} mm Hg range. It was noted that this could be caused by 1) vibrations induced into the ionization gage and leads, 2) the opening of microcracks, possibly to the outside of the system, 3) gas desorption from the chamber walls produced by the impact, or 4) gas release from the newly

formed silicate surfaces.

In this quarter it has been found that vibration of the ionization gage and leads does give a spurious pressure rise indication, as expected, but that this is easily recognizable due to the very rapid decrease of the pressure indication to its previous value after vibration ceases. Most of the pressure surges indicated this behavior to a degree in that the initial sharp rise was followed by an immediate drop to an intermediate pressure, but subsequently the pressure dropped slowly indicating that one or more additional processes were acting.

We then investigated whether the pressure bursts could be produced without actual cleavage. This was done by impacting the cleavage device while not in contact with the sample, and by impacting the vacuum system at various spots. We found that on almost every occasion pressure bursts were produced which were qualitatively identical to those produced during cleavage. From this it was concluded that the sample was not responsible (this does not mean that gas could not be coming from the sample, but rather that if it is, it is obscured by other processes).

The various system components were then helium leak tested while being impacted. No indications of a micro-leak were found. It appears hence that the most likely explanation is that the pressure bursts are caused by vibration-induced desorption from the chamber walls.

In order to reduce this problem, we have modified the cleavage technique so that rather than impacting the cleavage device we apply a gradually increasing pressure to it, thereby wedging the sample apart. With this modification, we are generally able to keep the pressure from rising above the mid 10^{-10} mm

Hg range /Note: for Run #46, in progress at this writing, no pressure burst occurred/.

It appears that at some time in the future a residual gas analyzer should be incorporated into the system to determine the types of gas being released during the bursts.

3.2 Copper Contamination

We have found on occasion that copper flakes have been deposited on the sample surfaces. It has been determined that these came from the top copper gasket and probably are deposited while the gasket seal is being opened to remove the samples. However, to reduce any uncertainty as to time of deposition, we have begun cleaning the gasket seat after each run. Should this not prove to be sufficient, we will modify the chamber so that the samples are shielded from line-of-sight exposure to the gasket.

3.3 Adhesion Measurements

Seven successful vacuum cleavage runs were made during this quarter. The pertinent experimental conditions are given in Table I.

Run #39 Cleavage Along Labradorite (001) Plane

After cleavage the upper sample rotated 30-40°. The cleavage surface was poor in that a number of noticeable steps were present with one relatively large prong sticking up from the edge of the lower sample. The samples were then brought into contact; the alignment was poor in that the upper sample was displaced from the lower sample with the apparent contact area being only about one fifth the total sample area. The first measurement of adhesion was made four minutes after cleavage and resulted in a force of 0.9 gms. Only a slight indication of a long range force was detected, and its magnitude was

slightly below measurement capability (<0.1 gm). The alignment was then improved and further adhesion measurements were made. The adhesion force was found to decrease rapidly with time, leveling off at 0.4 gms after about 10 minutes. Attempts were made to apply a load force in order to measure the load dependence of the adhesion. These attempts were unsuccessful due to the irregularity of the cleavage surfaces. The system was maintained at vacuum for 167 hours after cleavage during which time only a slight further decrease in adhesion magnitude occurred. All indications of adhesion disappeared upon admission of nitrogen to the system.

The data obtained are presented in Figure 1.

Run #40 Cleavage Along Microcline (001) Plane

After cleavage the upper sample rotated about 15°. Prior to recontact with the lower sample, the upper sample inadvertently contacted the copper base plate. It adhered strongly to this, but no measurement of the adhesion magnitude was obtained. First sample contact was made 20 minutes after cleavage. The adhesion force was 0.1 gm, and there were no indications of a long range force. This adhesion remained constant over the next four hours, at which time the run was terminated. Microscopic studies showed the presence of copper flakes adhering to high spots on both samples.

Run #41 Cleavage Along Andesine (001) Plane

After cleavage the upper sample rotated about 40° and within one minute recontacted the lower sample. The cleavage surfaces had several large steps on them so that contact was poor. The first measurement of adhesion gave an adhesion force of 0.9 gm. The force decreased over the next 20 minutes to about 0.3 gm. It thereafter decreased very slowly to a value of about 0.1 gm over a period of 42 hours. Only very slight indications of a long range force

were detected. All indications of adhesion disappeared upon admission of nitrogen to the system.

The data obtained are presented in Figure 2.

Run #42 Cleavage Along Labradorite (001) Plane

After cleavage the upper sample rotated about 40° and recontacted the lower sample within one minute. The cleavage surfaces were the poorest of any produced to date, being very irregular and having more the appearance of fractured surfaces. Contact between the two surfaces was solely at a raised area near the center of the lower sample, and the surfaces over the greater part of their area were separated by several tenths of a millimeter. Initial measurement of adhesion, about 7 minutes after cleavage, gave an adhesion force of about 0.6 grams. Only a very slight indication of a long range force was detected, and its magnitude was below measurement capability. The adhesion rapidly decreased to below detectable in about 40 minutes. During this time the cleavage device was brought into the vicinity of the upper sample to see whether the sample would be attracted to it (such attraction had been noted in previous runs, reported in the last quarterly report). No attraction was noted.

The data obtained are presented in Figure 3

Run #43 Cleavage Along Labradorite (001) Plane

After cleavage the upper sample rotated about 45° and recontacted the lower sample within one minute. The first measurement of adhesion, made 18 minutes after cleavage, gave a force of 1.5 gm. This decreased to 1.0 after 8 minutes. The lower sample was then rotated so that it was about 5° from atomic match in orientation with the upper sample. The adhesion force immediately increased to

about 2.8 gm. A distinct long range attractive force was then noted, which was sufficiently strong to bring the samples into contact at separations less than about 1 mm. The chisel was brought to the upper sample; no attraction of the sample to the chisel was detected. It was also noted that for the 5° alignment the upper sample, as it approached the lower sample, would rotate into what appeared to be atomic match (0°) as it was pulled into contact.

The lower sample was then rotated into various positions of atomic mismatch in orientation and it was found that upon doing this, the magnitude of the adhesion force decreased somewhat, and no indication of a long range force could be detected. The samples were then rotated back to the 5° orientation and the resultant observations were the same as reported previously. The system was maintained at vacuum for about 330 hours after cleavage, during which time the adhesion force decreased slightly. The data up to T = 330 hours are presented in Figure 4.

At T = 330 hours the ion pump was turned off, allowing the system pressure to rise slowly. In 30 minutes the pressure had risen to 9×10^{-9} mm Hg. The adhesion force was much smaller, as was the long range force. The pump was then turned on, the system pumped to 1.8×10^{-10}, and the adhesion measured. It was found that both the adhesion and long range force were still small. However, within 20 minutes the adhesion force had increased to its previous value, whereas the long range force remained small. The pump was then cycled a second time with similar results except that the adhesion did not recover. Finally, the pump was turned off again and small bursts of nitrogen were admitted to the system. Detectable, but small, adhesion remained to a pressure of 10^{-4} mm Hg. At this point the system was let up to atmospheric pressure and all indications of adhesion disappeared. The data obtained are presented in

Figure 5.

Run #44 Cleavage Along Labradorite (001) Plane

Following cleavage the upper sample rotated about 45°. The first measurement was obtained about 12 minutes after cleavage; the adhesion force was about 0.8 gm. The next measurement was not made until 20 hours later due to experimental difficulties and the adhesion force was 0.2-0.3 gm at that time. The force decreased slowly thereafter, falling below measurement capability after about 70 hours. Some indications of a very weak long range force were detected during the early stages of the run.

Run #45 Cleavage Along Labradorite (001) Plane

Following cleavage the upper sample rotated about 30°. The first adhesion measurement was made 5 minutes after cleavage, giving a force of 0.3 gm. A significant long range force was present. The adhesion force decreased slowly to 0.1 gm over a period of 55 hours, at which time the experiment was terminated. The long range force remained moderately strong for about 1 hour after cleavage. Thereafter, it decreased rapidly to barely detectable. The data obtained are presented in Figure 6.

Figure 7 presents a summary of the data from all runs, plotted to a common scale.

4.0 DISCUSSION

In the previous quarterly report, it was concluded that

a. More than one process is acting to produce the observed adhesion between the vacuum produced surfaces.

b. The higher magnitude adhesion appearing initially after cleavage is possibly caused by the action of the normal atomic bonding forces, but that surface electrostatic charging is definitely active.

The findings during this quarter tend to substantiate these conclusions, though it is evident that many more data are required.

4.1 Type of Force Acting

Three types of force can act to produce adhesion between vacuum-formed surfaces. These are: the normal atomic bonding forces, the dispersion forces, and surface electrostatic charging-produced forces.

From the data obtained to date, it is concluded that surface charging is indeed acting. The evidence for this is the presence, in most cases, of an attractive force whose range of effectiveness greatly exceeds that of either of the other possible forces.

The behavior of this charging has been found to be highly variable. At times, it has appeared to be quite strong; in other instances, it was very weak or undetectable. In some cases, the surfaces appeared to have a significant net charge, being attracted to any metal in the vicinity; in other instances, no such attraction was detectable. For one run, the charge seemed to have a distinct geometric arrangement on a macroscale in that it would physically rotate the upper sample into "atomic" alignment with the lower sample.

In the previous report it was noted that these charges are probably produced through breakage of the atomic bonds with the resultant charge separation, and that this could result in either a random or non-random charge distribution. If random, the magnitude of the resultant adhesion would vary greatly for runs

involving the same material. This has been found to be the case. However, the precise nature of the causal agent is less clear now than it was at the time of the previous report. The reasons for this increased uncertainty are given below.

The surfaces of the cleaved samples have been carefully studied and it has been found that permanent stress deformation is present in every case. There are two main stressed areas, one at the point of cleavage and one opposite this point. A third, secondary stressed area is often found to one side or the other of the cleavage notch. This area appears to be due to variations (non-symmetry) in inserting the chisel into the notch. If the hypothesis that dislocations can carry their charge with them is correct, then the observed stress patterns would suggest that dipole, quadrapole, and/or hepapole charge fields may form on the cleavage surface. This could account for the rotational and translational orientation force observed during Run #43. In addition, it had been concluded previously that piezoelectric effects could not be responsible for the charge production since all minerals used possess a center of symmetry. However, it is possible that with the strains present during cleavage a second order piezoelectric effect could produce a polarization which becomes permanent, across the interface, after cleavage.

With the present uncertainties as to the cause(s) of the surface charging, it appears evident that an electrometer-probe should be inserted into the system as soon as feasible to plot the charge distribution.

It was concluded in the previous quarterly report that the normal atomic bonding forces may be contributing to the adhesion. The only reasonably direct information about this was the finding of material transfer to the

upper sample after it contacted the metal bucket (Run #28 of previous report). All the other observations were indirect. These included the noted rapid decrease in adhesion force with time compared to the constancy of the long range force over this same time period; and the considerable surface roughness for some of the runs (indicating that dispersion forces were not primarily responsible).

Additional information obtained during this quarter, also indirect, is: 1) Runs of considerably different surface roughnesses and strength of the long range force showed similar adhesion magnitude, and 2) Run #43, where the system pressure was varied, showed that the adhesion force recovered the first time the system was returned to 10^{-10} mm Hg while the long range force did not.

It is evident that many more data are required before these uncertainties can be resolved. In particular, as noted earlier, an electrometer-probe should be inserted into the system to obtain information regarding the charge distribution. Also, the spring measuring device should be replaced by a rigid strain gauge mounting so that the force-distance profile for the long range force can be determined. Finally, means should be incorporated into the system for discharging the surfaces so that the contribution to the adhesion of the normal atomic and/or dispersion forces can be separated from the charging effect.

4.2 Time After Cleavage, and Pressure Effects

The pertinent data for time after cleavage effects are shown in Figures 4 and 7. In general, the force of adhesion decreases rapidly during the first 20 minutes. Thereafter, it decreases very slowly over the duration of the run (maximum run length = 330 hours). Between measurements the samples are

kept separated, and hence are exposed to the ambient environment. The minimum time for monolayer formation at the experimental pressure is about 3 hours. Hence, it is seen that the adhesion is relatively insensitive to the gradual accumulation of adsorbed gas atoms on the surface.

The effect of ambient pressure on adhesion force is shown in Figure 5. It is seen that the adhesion magnitude decreased rapidly when the system pressure was increased to 10^{-8} mm Hg. Upon re-evacuation to 10^{-10} mm Hg, it recovered in about 20 minutes. Taking the system to 10^{-8} mm Hg caused a second sharp drop. The adhesion force, however, did not recover with the second re-evacuation. Subsequently, measurable adhesion remained until air was admitted to the system from a pressure of 10^{-4} mm Hg. The persistence of some degree of adhesion into the 10^{-4} mm Hg range for this run is explainable on the basis of dispersion forces since, as no rotation occurred, the sample surfaces were well matched. It may also be explainable on the basis of surface charging, but this latter possibility cannot be evaluated until we arrive at a better understanding as to the cause and nature of this charging.

4.3 Gas Bursts Versus Magnitude of Adhesion

The data from Table I and Figure 7 show generally that the larger the gas burst, the lower the magnitude of the initial adhesion. This indicates a definite contamination effect and hence larger magnitude adhesion may be observed if the gas bursts can be eliminated.

5.0 CONCLUSIONS

The following conclusions for the vacuum formed surfaces apply at this time:

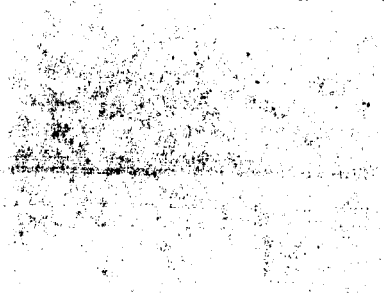
a. More than one type of process appears to be responsible for the observed adhesion.

b. One of these is surface electrostatic charging; the cause of this charging is not known; it may be associated directly with breakage of atomic bonds across the interface, or it may be the result of stresses produced during cleavage.

c. The normal atomic bonding forces may be active, particularly within the first hour after cleavage.

d. Dispersion forces probably make a negligible contribution to the adhesion, except possibly for those runs at 0° orientation.

e. Further studies should include the installation of an electrometer to map the surface charge, a rigid force measuring mount to obtain force-distance plots, and a means for discharging the surfaces.



REFERENCE

Ryan, J. A., Experimental Investigation of Ultra-High Vacuum Adhesion as Related to the Lunar Surface, Eighth Quarterly Progress Report and Second Year Summary, Douglas Report DAC-59288, 1966.

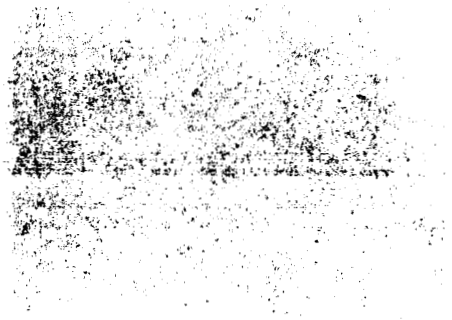


Table I

EXPERIMENTAL CONDITIONS FOR VACUUM FORMED SAMPLES

Run No.	Sample Type	Cleavage Plane	Pressure (mm Hg)	Comments
39	Labradorite	(001)	1.2×10^{-10}	Pressure burst to 5×10^{-10} mm Hg
40	Microcline	(001)	1.9×10^{-10}	Pressure burst to 2×10^{-9} mm Hg
41	Andesine	(001)	3.3×10^{-10}	Pressure burst to 1×10^{-9} mm Hg
42	Labradorite	(001)	1.7×10^{-10}	Pressure burst to 4×10^{-9} mm Hg
43	Labradorite	(001)	2.3×10^{-10}	Pressure burst to mid 10^{-10} range
44	Labradorite	(001)	2.9×10^{-10}	Pressure burst to mid 10^{-10} range
45	Labradorite	(001)	2.0×10^{-10}	Pressure burst to 8×10^{-9}

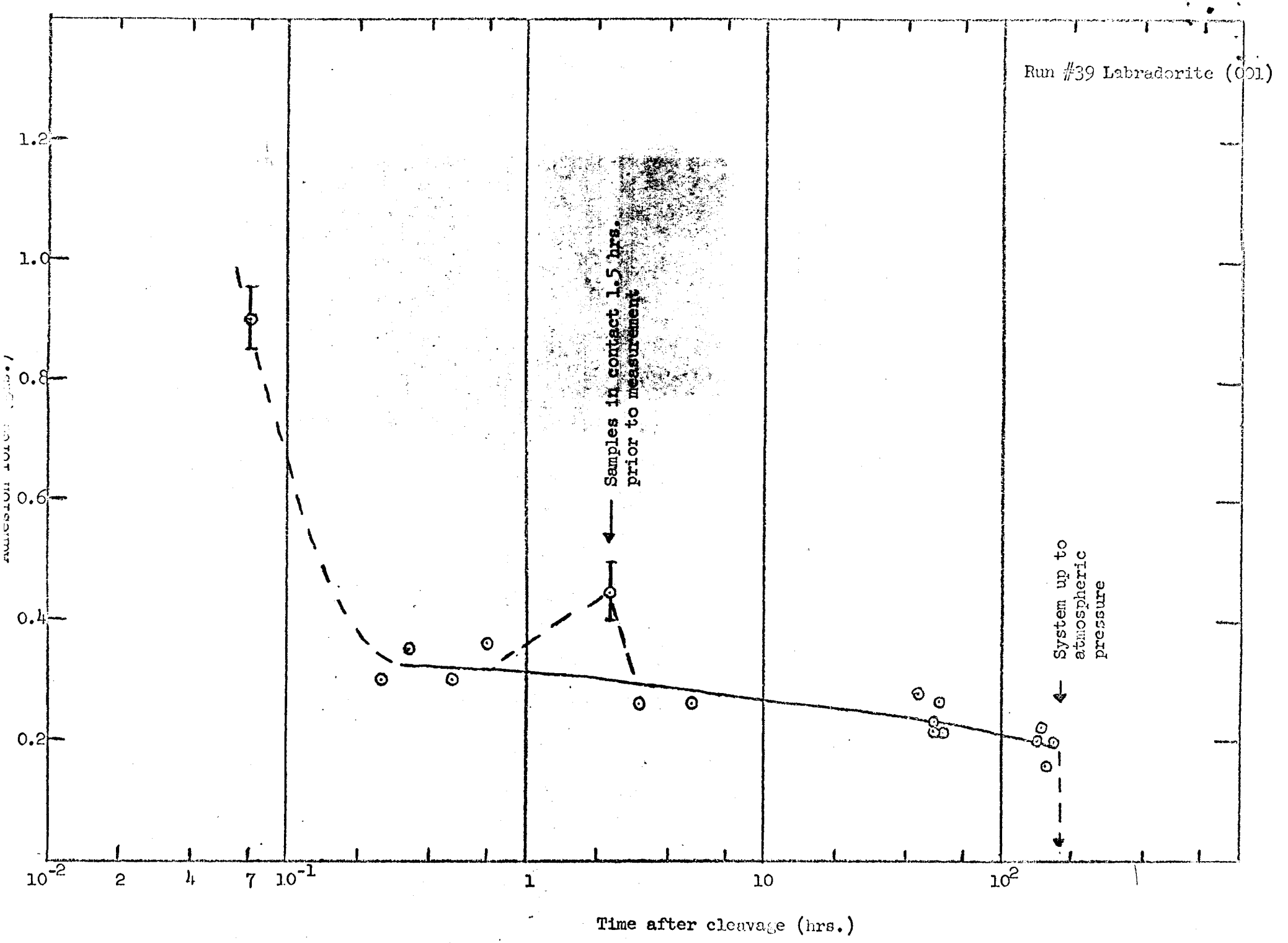

Run #39 Labradorite (001)
1.2
1.0
0.8
0.6
0.4
0.2
Samples in contact 1.5 hrs. prior to measurement
System up to atmospheric pressure
10^{-2}
2
4
7
10^{-1}
1
10
10^{2}
Time after cleavage (hrs.)

Figure 1

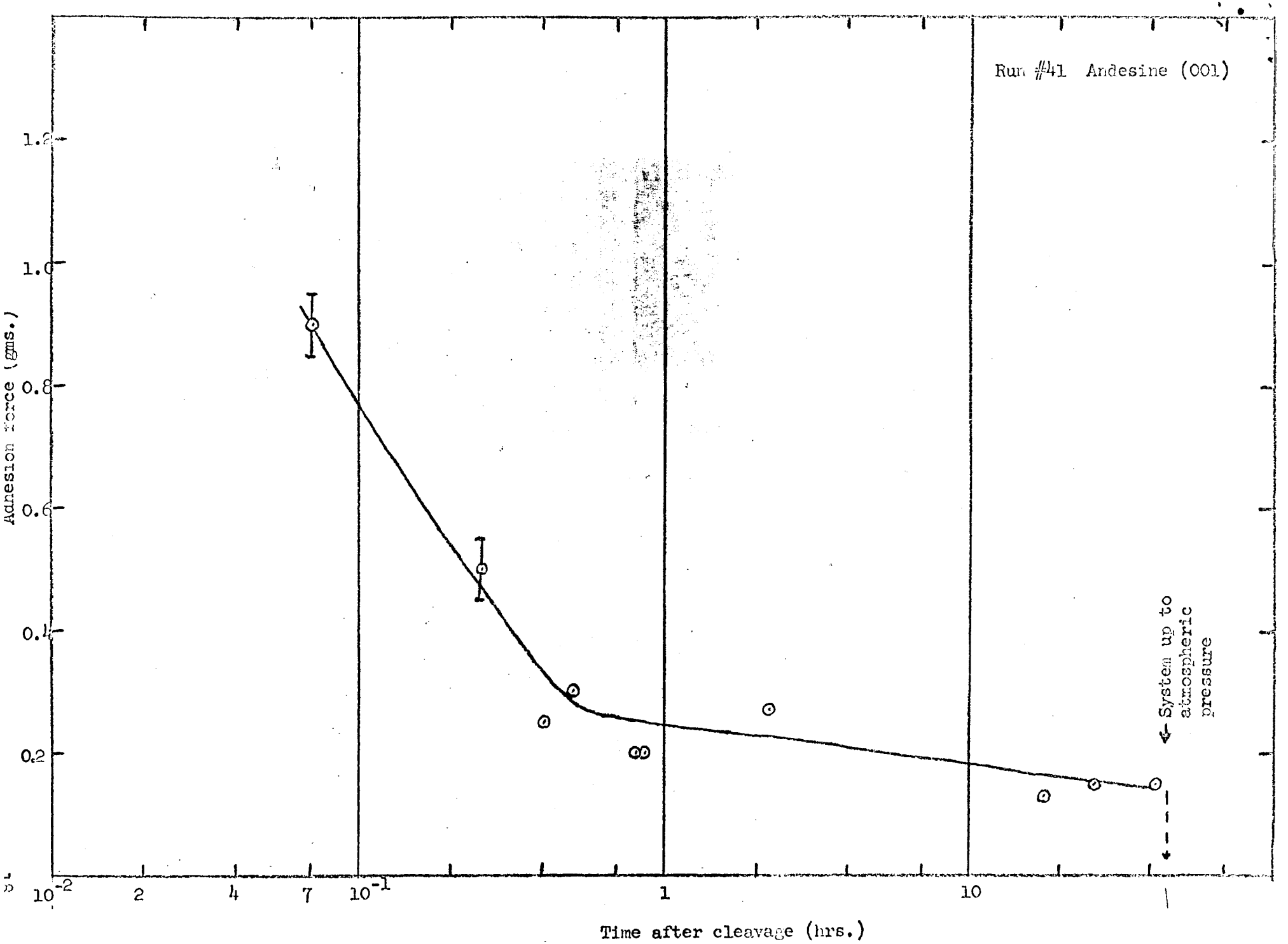

Run #41 Andesine (001)
1.2
1.0
0.8
0.6
0.4
0.2
Adhesion force (gms.)
10^{-2}
2
4
7
10^{-1}
1
10
System up to atmospheric pressure
Time after cleavage (hrs.)

Figure 2

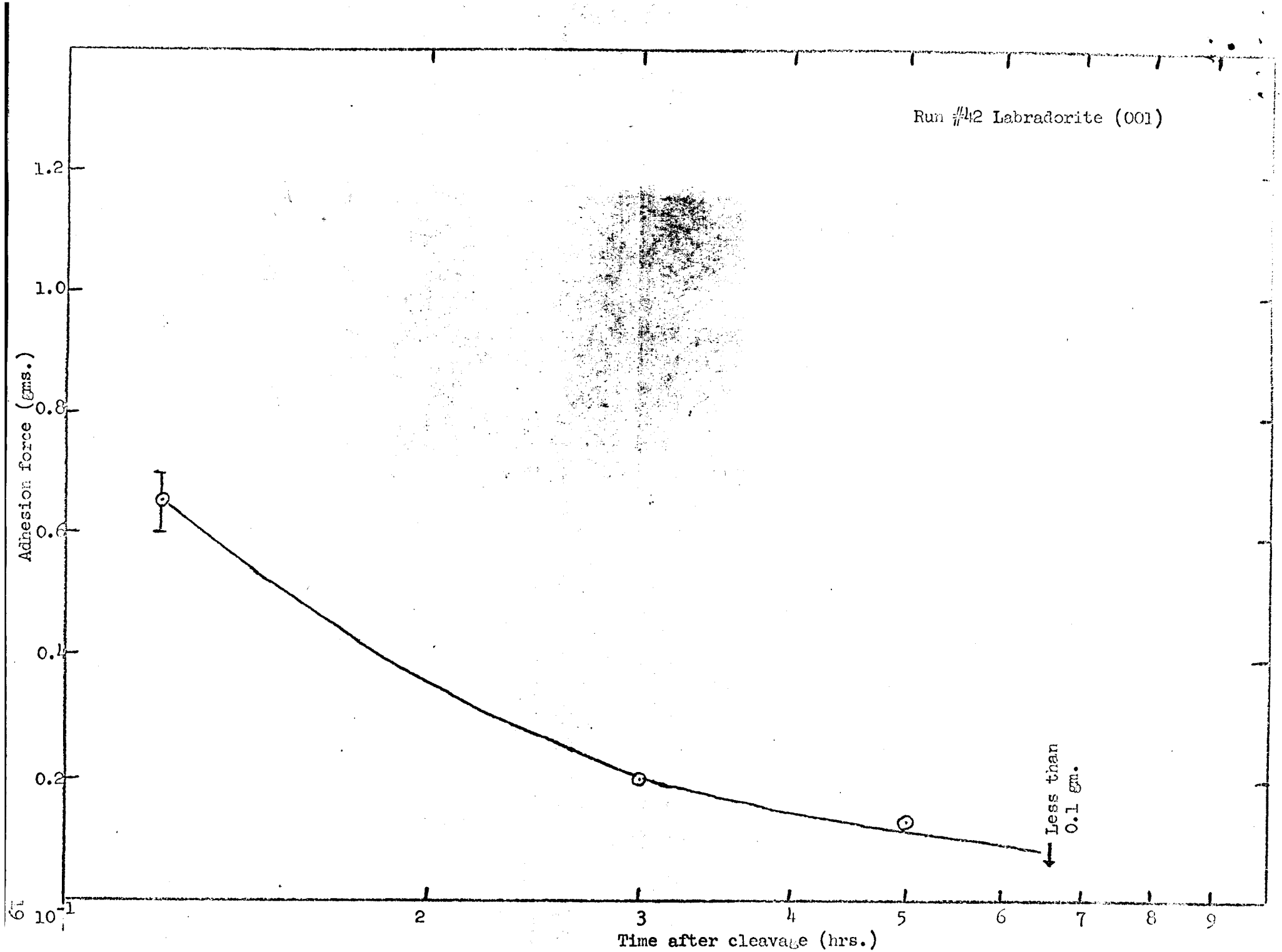

Run #42 Labradorite (001)
Adhesion force (gms.)
1.2
1.0
0.8
0.6
0.4
0.2
10^{-1}
2
3
4
5
6
7
8
9
Time after cleavage (hrs.)
Less than
0.1 gm.

Figure 3

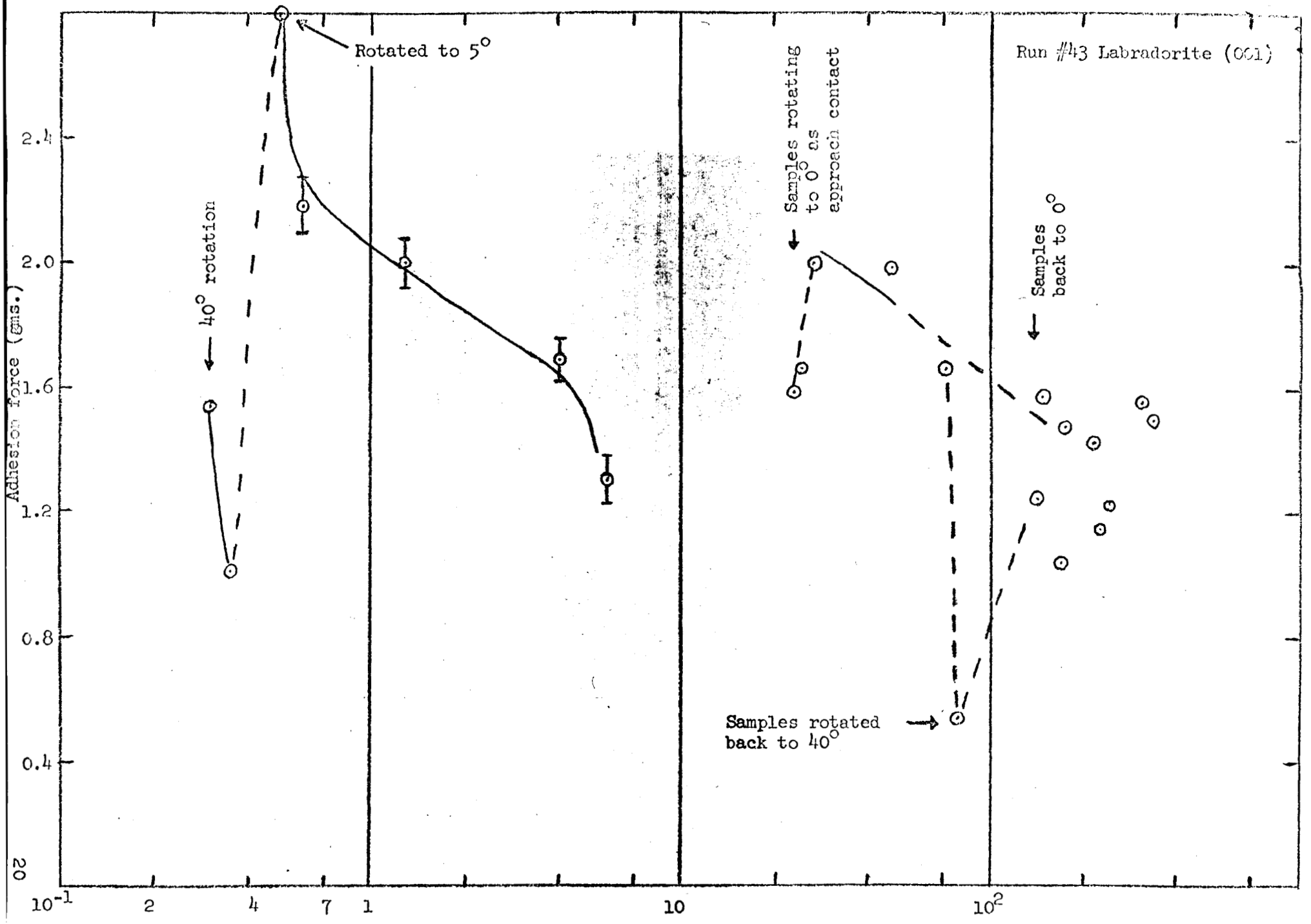

Run #43 Labradorite (001)
Rotated to 5°
40° rotation
Samples rotating to 0° as approach contact
Samples back to 0°
Samples rotated back to 40°
Adhesion force (gms.)
2.4
2.0
1.6
1.2
0.8
0.4
20
10^{-1}
2
4
7
1
10
10^{2}
Time after cleavage (hrs.)

Figure 4

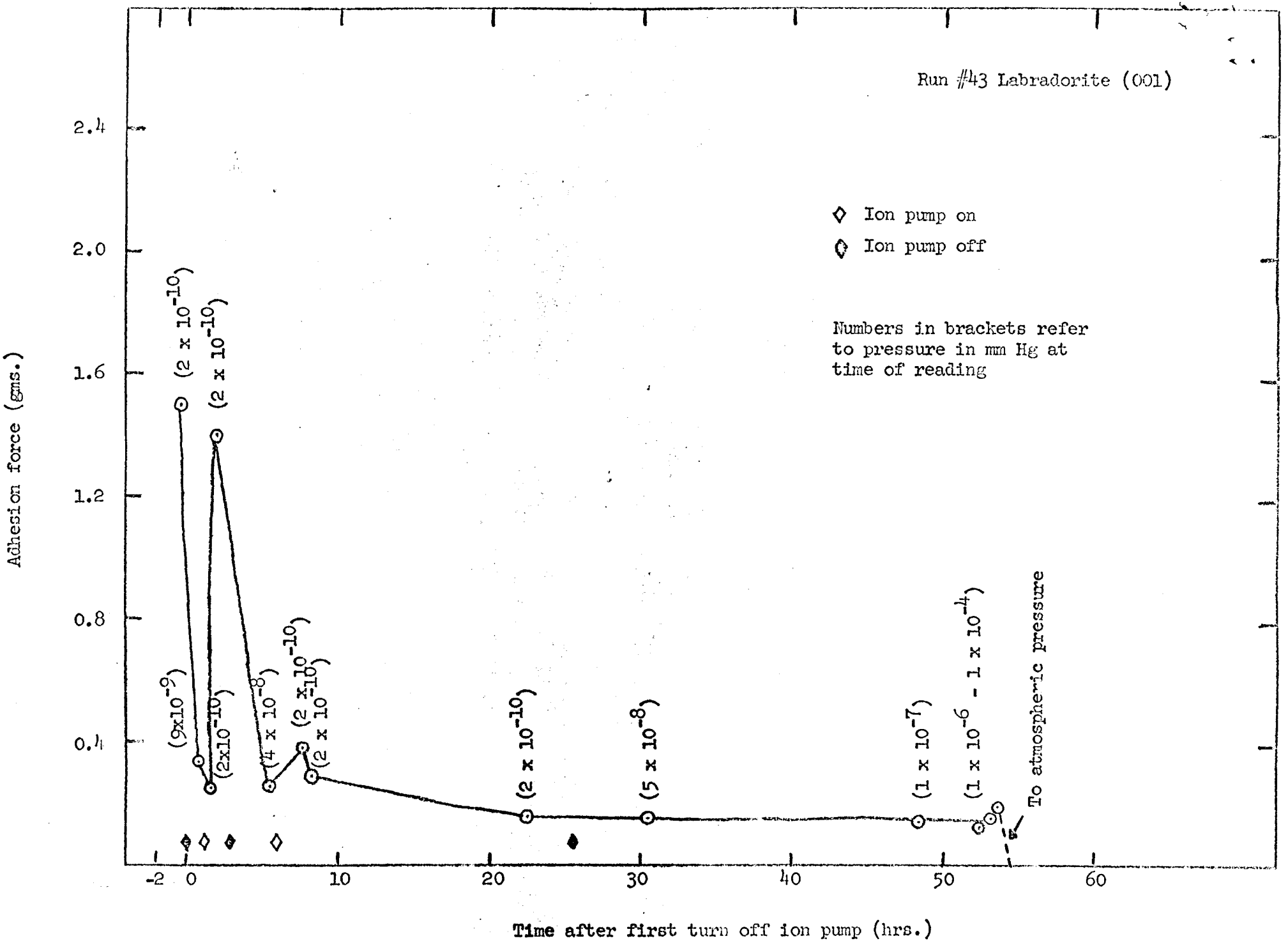

Run #43 Labradorite (001)
Ion pump on
Ion pump off
Numbers in brackets refer to pressure in mm Hg at time of reading
Adhesion force (gms.)
2.4
2.0
1.6
1.2
0.8
0.4
(2 x 10⁻¹⁰)
(2 x 10⁻¹⁰)
(9x10⁻⁹)
(2x10⁻¹⁰)
(4 x 10⁻⁸)
(2 x 10⁻¹⁰)
(2 x 10⁻¹⁰)
(2 x 10⁻¹⁰)
(5 x 10⁻⁸)
(1 x 10⁻⁷)
(1 x 10⁻⁶ - 1 x 10⁻⁴)
To atmospheric pressure
-2
0
10
20
30
40
50
60
Time after first turn off ion pump (hrs.)

Figure 5

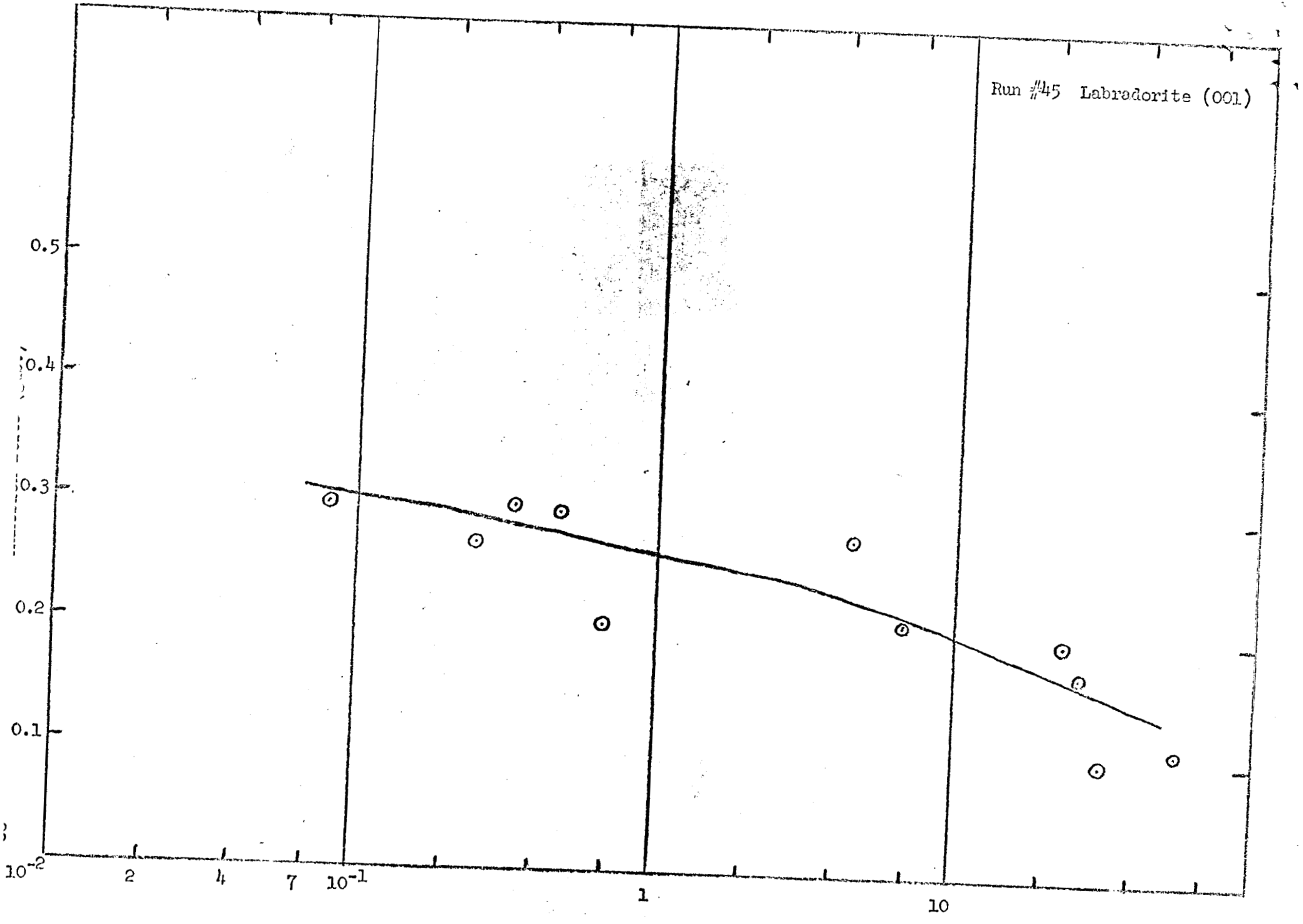

Run #45 Labradorite (001)
0.5
0.4
0.3
0.2
0.1
10^{-2}
2
4
7
10^{-1}
1
10
Time After Cleavage (hrs.)

Figure 6